Scientific Research Publishing

Journal of Materials Science and Chemical Engineering, 2020, 8, 1-10
https://www.scirp.org/journal/msce
ISSN Online: 2327-6053
ISSN Print: 2327-6045

In Situ Synthesis of Titanium Carbide in Pure Aluminium

Satish Babu Boppana

Department of Mechanical Engineering, School of Engineering, Presidency University, Bengaluru, India
Email: satishbabu3@yahoo.co.in

How to cite this paper: Boppana, S.B. (2020) *In Situ* Synthesis of Titanium Carbide in Pure Aluminium. *Journal of Materials Science and Chemical Engineering*, **8**, 1-10.
https://doi.org/10.4236/msce.2020.81001

Received: November 30, 2019
Accepted: December 27, 2019
Published: December 30, 2019

Abstract

The present work reports on the mechanism of formation of TiC in pure Aluminium melt. A halide salt of Al_3TiF_6, graphite powder and pure Al were used to prepare *in situ* Al-TiC Metal Matrix Composites (MMCs). Scanning Electron Microscopy (SEM) and Energy Dispersive Spectroscopy (EDS) were used to determine the microstructural characteristics of the composite. XRD was further used to determine the phases involved in the composite for confirming the formation of TiC. Further to assess the mechanism involved in the *in situ* reactions, DTA/TGA thermograms were used to analyze the reactions between molten Al, halide salt and graphite powder. Tensile tests were conducted to study the fracture behavior of the *in situ* prepared MMCs.

Keywords

In Situ, TiC, Microstructure, Tensile Strength, Fracture Analysis

1. Introduction

With the ever increasing commercial applications of particulate reinforced composites, studies are carried out to find prospective ways of different processing methods of composites [1]. During processing such composites, a number of ceramic phases can be included as particulate reinforcements in Aluminium Metal Matrix Composites (AMCs) [2]. Some of these include Al_2O_3, TiO_2, SiC, B_4C, BN, TiB_2, Si_3N_4, AlN. A fine and ultra-grain structure usually results in improved mechanical properties and owing to the grain refining efficiency of Al-TiC, a number of researchers have studied on different techniques to synthesize the composite [3] [4].

Titanium Carbide (TiC) particulates offer good wettability with Aluminium. The other reasons to consider TiC are due to its high temperature stability, high hardness, low weight and low chemical reactivity [5]. Among the several me-

thods of preparing TiC reinforced composite, like liquid-solid reactions, liquid-liquid reactions, reaction gas injection, exothermic dispersion and self propagating high-temperature synthesis, the *in situ* method of preparing the composite is becoming popular due to homogeneous distribution of reinforcement throughout the matrix [6]. TiC reinforced-aluminium composites were also produced by adding elemental carbon to Al-Ti molten alloy, while observing that a temperature of 1200˚C was responsible for the formation of Al_3Ti particles along with TiC. However, increasing the temperatures by 100˚C would reveal only TiC phases [7]. Al-TiC composite was also fabricated through powder metallurgy route by using elemental powders of Ti, C and Al through compaction. Spherical TiC particles were observed in the matrix; however Al_3Ti phases were also seen [8].

Yucel Birol [9] worked on the thermal response of composites prepared by using Al-Ti and graphite powder through milling and blending. Differential Thermal Analyzer (DTA) scans on the composites exhibited endothermic and exothermic peaks. The X-ray Diffractometer (XRD) patterns had also shown the presence of Al_3Ti and Al_4C_3 phases and could have also influenced the thermograms.

The mechanical behavior of TiC reinforced aluminium based metal matrix composites processed with the addition of TiC particles in molten Aluminium (99.7% Al) was investigated by A. E. Karantzalis *et al.* [10]. Some of the castings were extruded to rods, while others were used in as-cast condition. The Ultimate Tensile Strength (UTS) of the specimen increased with the increasing reinforcement content and attributed to grain refining process, while X.C. Tong [11] investigated on the tensile fracture surfaces of Al-TiC composites processed through ingot metallurgy by melting a mixture of Al, Ti and graphite powder of the size of 45 microns in an induction furnace under a cover of argon gas. A ductile mode of fracture was observed during the tensile tests while the strengthening mechanism was affirmed to the micromechanics approach.

Several researchers have been investigating to know the microstructure and property particularly at the interface site [12]. Since the Al-Ti-C system is gaining popularity due to its relevance as grain refiners, researchers have been trying to continuously predict the reactions occurring through ternary phase diagrams [13] [14] [15] [16] [17], but very little has been analyzed on the formation of TiC at higher temperatures. Hence an attempt has been made to study the effect of temperature on the *in situ* formation of reinforced particle.

2. Experimental Work

2.1. Preparation of Composites

The related work involves preparing Al-TiC MMCs using an *in situ* technique. A measured quantity of K_2TiF_6 halide salt and graphite powder, according to a known stoichiometric ratio was taken in an Aluminium foil and preheated to drive off any moisture present, using a muffle furnace at a temperature of 300˚C.

Pure Aluminium in the form of ingots were pickled to remove impurities present using NaOH solution and further cleaned by using nitric acid and methanol. A calculated amount of ingots corresponding to Al-5 wt% TiC were then placed in a SiC crucible and melted at 660˚C and super heated to reach 800˚C. Halide salt and graphite powder were then added into the melt. A chromel-alumel thermo couple was used to control the temperature digitally with an accuracy of ±5˚C. A cover flux was poured into the crucible to prevent oxidation of liquid aluminium. Hexachloro ethane tablets were then added to the melt, to remove hydrogen gas formed during the reaction. The melt is carefully stirred at regular intervals with a graphite rod until the reaction completes for 30 min. The spent salt is decanted from the surface of the melt, and the melt is finally poured into a preheated permanent mould. The molten material was poured into the dies at temperatures of 800˚C and 930˚C. After solidification, the castings were removed.

2.2. Thermal Analysis

The composite was subjected to Perkin-Elmer Simultaneous Thermal Analyzer (TGA/DTA) cell. The scan rate in the unit was fixed at 10˚C/min and the gas flow rate was fixed at 80 ml/min. The area under the DTA peak is chosen to calculate the enthalpy change for finding out the transformations that have taken place during melting. The weight changes of the sample were recorded during the test and the TGA thermograms were recorded.

2.3. Microstructural Characterization

A specimen of the size of about 8mm was cut from the bottom of the casting using hack saw machine. Using a series of rough and smooth files, it was polished followed by working on a belt grinder, to remove burrs if any. Polishing was again carried out using emery sheets of increasing fineness. Finally a disc polisher with a velvet cloth and Al_2O_3 solution was used in order to get a mirror finish. The specimen was etched with Kellers reagent after being washed with distilled water. Image Analyzer, SEM equipped with EDS and X-Ray Diffractometer were used to study the microstructure.

2.4. Tensile Test

In order to calculate the ultimate tensile strength and elongation, a 40 ton capacity servo controlled universal testing machine was used. The tensile specimen was prepared to follow the ASTM E8 standards. An extensometer was attached coinciding with the axis of the specimen. Load was applied gradually while simultaneously noting down load and strain at room temperature.

3. Results and Discussion

3.1. DTA/TGA Analysis

The thermogram (**Figure 1**) shows two exothermic peaks starting at 657˚C and

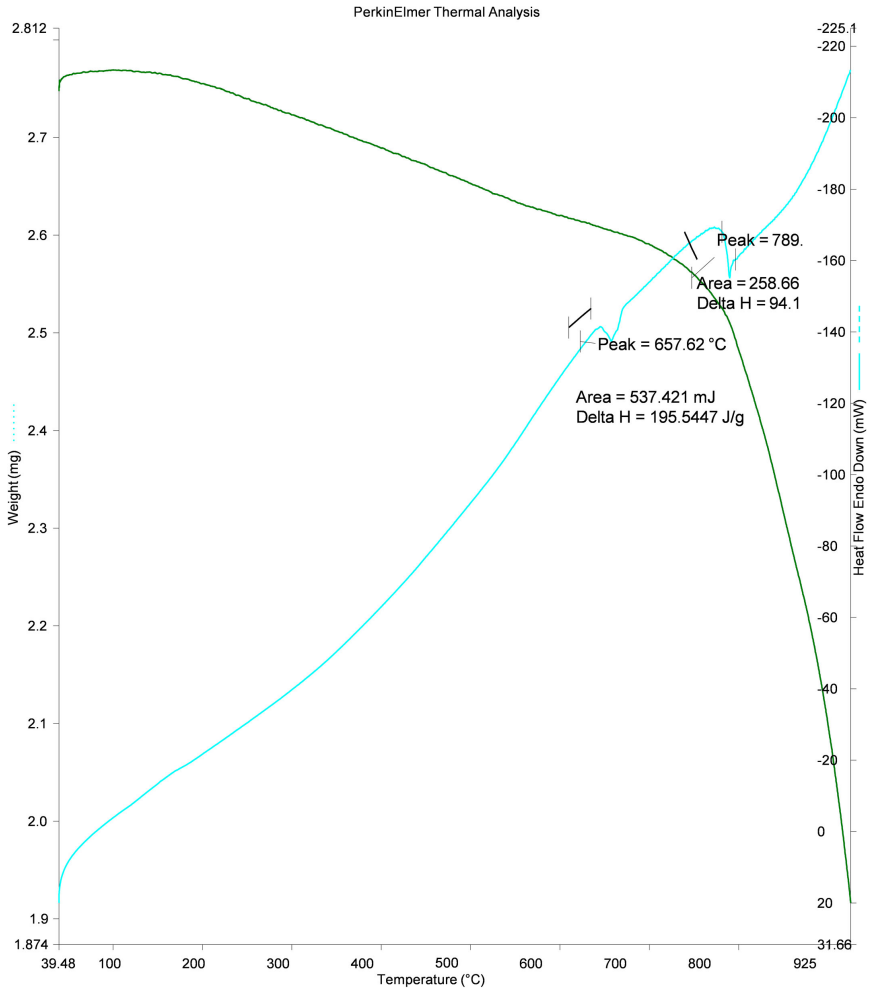

Figure 1. Thermogram of Al-TiC composite depicting the transformations during heating.

ending at 670˚C while another peak starting at 789˚C and ending at 795˚C. The two peaks reveal the melting of pure Aluminium ingots and simultaneous formation of Al_3Ti and TiC phases.

The mass of the sample while heating is recorded through a TGA curve. From **Figure 2**, it is observed that the curve starts to move in the downward direction beyond 670˚C, indicating melting of pure Aluminium, while at 795˚C, formation of Al_3Ti and TiC phases takes place. The DTA and TGA curves relatively give the same data for the formation of *in situ* TiC.

Further after that, the curve gradually keeps coming down as the composite is heated with respect to time, revealing that the mass of the sample decreases with increasing temperature.

3.2. Microstructures of the Samples

The optical micrograph obtained through an Image Analyzer of the composite in **Figure 3**, reveals the presence of plate like Al_3Ti particles throughout the matrix. However grayish dark spots were also seen here and there in the matrix.

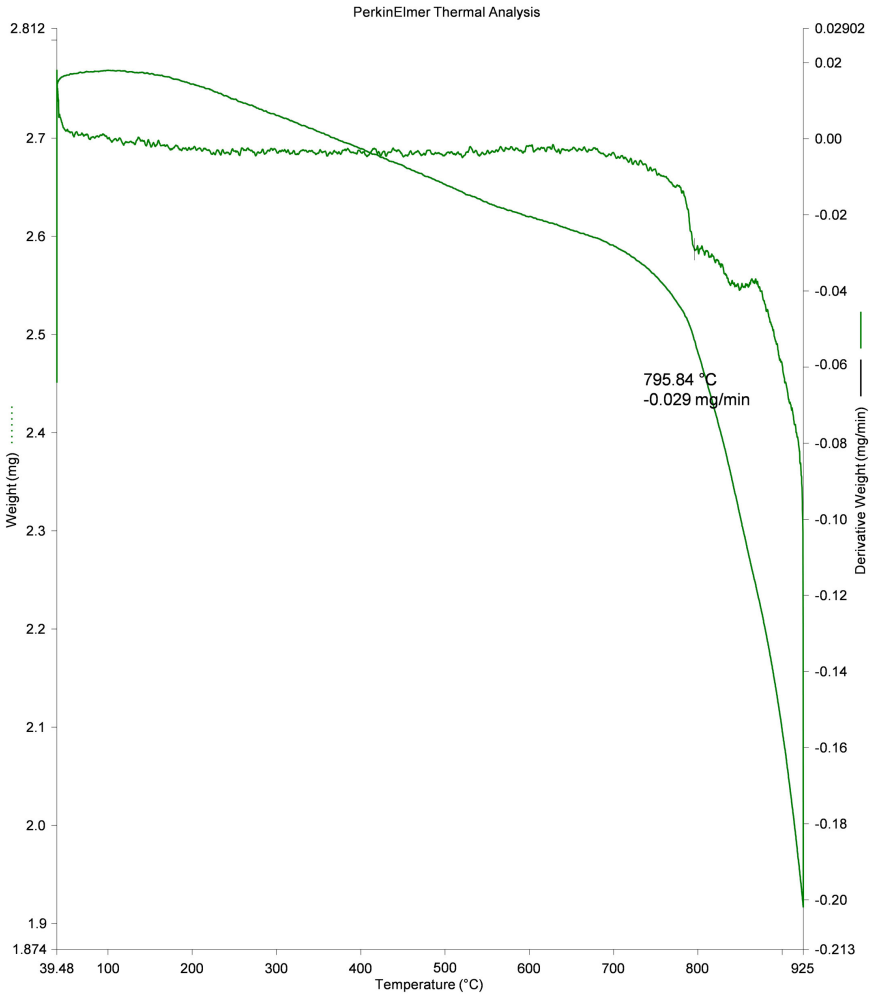

Figure 2. Thermogram measuring mass of the composite over time while heating.

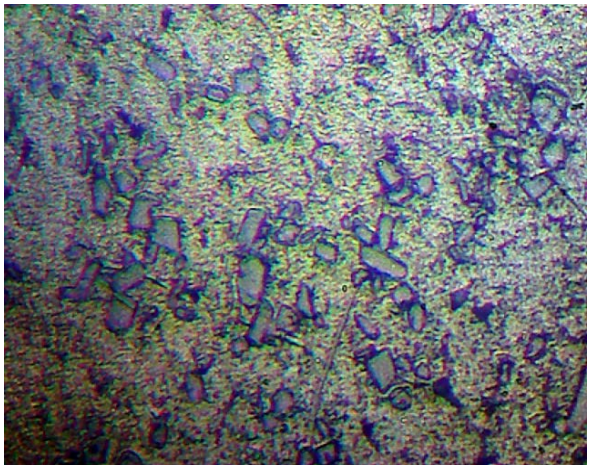

Figure 3. Optical micrograpgh of Al-5TiC composite processed at 800˚C.

The black spots that were found, might be due to excess carbon left out after reacting with Al_3Ti particles. A reaction temperature of 800˚C might not have been sufficient for the complete reaction to take place and hence Al_3Ti particles

are seen in more numbers. Upon increasing the temperature to 930˚C, almost all the aluminide particles converted to TiC. Figure 4 clearly indicates the spherical TiC particles scattered in the matrix. Henceforth, the composite processed at a reaction temperature of 930˚C was only considered for further characterization. In order to further investigate about the reinforcement at a magnification of 1000×, Scanning Electron Microscopy equipped with EDS was used. Figure 5 shows a TiC particle in a SEM micrograph, which is confirmed using Energy Dispersive Spectroscopy for elemental analysis through Figure 6.

In order to find out the phases involved, X-ray powder diffraction analytical technique was used. The peaks from Figure 7, suggested the cubic structure of Aluminum and traces of TiC throughout the matrix.

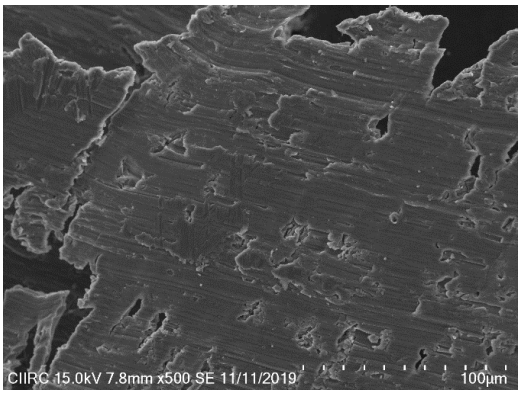

Figure 4. SEM micrograpgh of Al-5TiC composite processed at 930˚C.

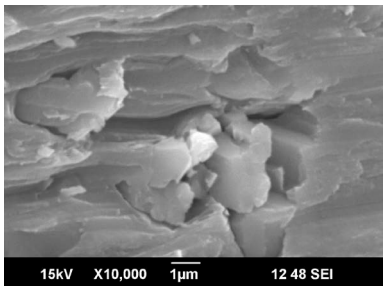

Figure 5. SEM micrograph of Al-5TiC composite.

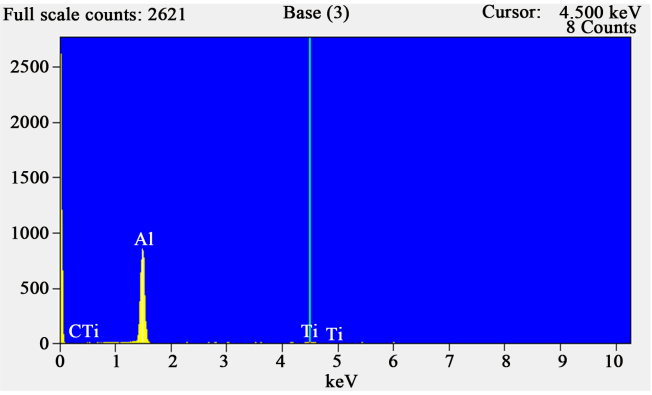

Figure 6. EDS analysis pattern of the composite.

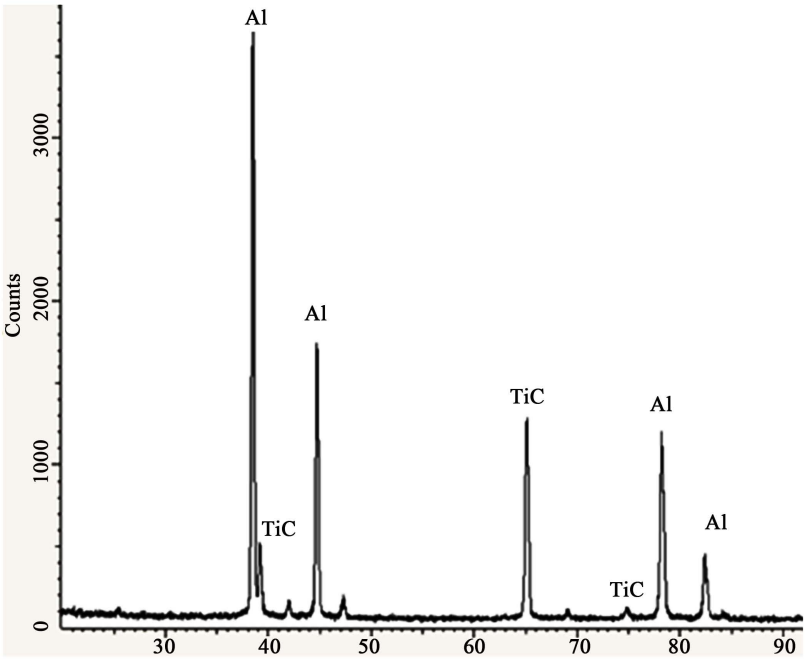

Figure 7. XRD spectrum of the composite.

3.3. Tensile Strength and Fracture Analysis

The Ultimate Tensile Strength (UTS) was recorded at 132 MPa while elongation was 9%. The increase in UTS compared to pure Aluminium is attributed to the presence of hard and dense intermetallic phases of TiC. Titanium acts as a grain refiner for the melt and promotes heterogeneous nucleation. Other reason for increase in strength is due to solid solution strengthening [8]. The sliding of grain boundary is altered by the presence of intermetallic phases, hence obstructs the dislocation movement.

The specimen showed a ductile fracture. It is seen clearly from the micrographs (**Figure 8** & **Figure 9**) that there is no disbonding between TiC and matrix. The fracture could have occurred through nucleation, growth and propagation [11].

Figure 8 shows the micrograph of fractograhic sample at the point of necking. A small number of TiC particles are observed to be holding back and clinging in the dimples produced during fracture. Due to the grain refinement by Ti particles, the particle size decreases. It is well known that as the particle size decreases, ductility always increases. From the micrograph (**Figure 9**) we are able to observe fine particles of TiC, which again supports ductile fracture. The size of reinforcement greatly affects the fracture mechanism. During the application of tensile load, when the specimen is about to fracture through the formation of pores, it is observed that the pore height grows in the direction of applied load. All the neighboring interconnecting pores start to grow in the same direction, thereby leading to interconnection between a number of pores. Again, this is clearly evident in the micrograph (**Figure 9**) with a number of adjacent pores leading to ductile failure and thereby reducing sudden failure.

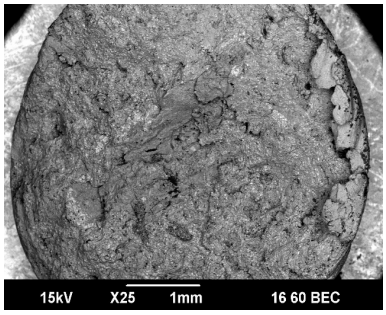

Figure 8. SEM fractograph at necking.

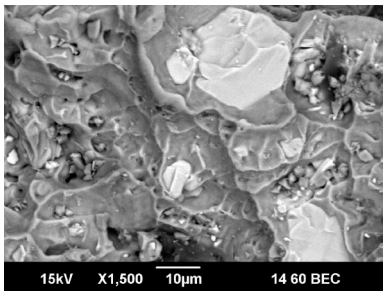

Figure 9. SEM fractograph showing dimples.

4. Conclusions

- Al-5TiC composites were successfully prepared by using a halide salt, graphite powder and pure Aluminium.
- Microstructural studies reveal the presence of spherical TiC particulates in the matrix; elemental and phase analysis through SEM-EDS, and XRD reflect the formation of TiC.
- DTA/TGA thermograms reveal the mechanism of reactions during the formation of the composite; exothermic reactions and endothermic reactions reveal the melting points during the formation of TiC.
- Fractographic analysis of the tensile specimen exhibits a ductile failure with necking and dimples at the point of failure.

Acknowledgements

The author would thank the Sophisticated Test and Instrumentation Centre (STIC), Cochin for providing SEM and TG/DTA facilities.

Conflicts of Interest

The author declares no conflicts of interest regarding the publication of this paper.

References

[1] Gowda, K.P., Prakash, J.N., Gowda, S. and Babu, B.S. (2015) Effect of Particulate Reinforcement on the Mechanical Properties of Al2024-WC MMCs. *Journal of Minerals and Materials Characterization and Engineering*, **3**, 469-476.

https://doi.org/10.4236/jmmce.2015.36049

[2] Lee, K.B., Sim, H.S. and Kwon, H. (2006) Reaction Products of Al/TiC Composites Fabricated by the Pressureless Infiltration Technique. *Metallurgical and Materials Transactions A*, **37**, 2517-2527. https://doi.org/10.1007/s11661-005-0125-0

[3] Boppana, S.B. and Chennakeshavalu, K. (2009) Preparation of Al-5Ti Master Alloys for the *In-Situ* Processing of Al-TiC Metal Matrix Composites. *Journal of Minerals and Materials Characterization and Engineering*, **8**, 563-568. https://doi.org/10.4236/jmmce.2009.87049

[4] Harsha, R.N., Kulkarni, V.M. and Babu, B.S. (2018) Severe Plastic Deformation: A Review. *Materials Today*: *Proceedings*, **5**, 22340-22349. https://doi.org/10.1016/j.matpr.2018.06.600

[5] Li, P., Kandalova, E.G., Nikitin, V.I., Makarenko, A.G., Luts, A.R. and Yanfei, Z. (2003) Preparation of Al-TiC Composites by Self-Propagating High-Temperature Synthesis. *Scripta Materialia*, **49**, 699-703. https://doi.org/10.1016/S1359-6462(03)00402-0

[6] Dayanand, S., Babu, S. and Auradi, V. (2018) Experimental Investigations on Microstructural and Dry Sliding Wear Behavior of Al-AlB_2 Metal Matrix Composites. *Materials Today*: *Proceedings*, **5**, 22536-22542. https://doi.org/10.1016/j.matpr.2018.06.625

[7] Kerti, I. (2005) Production of TiC Reinforced-Aluminum Composites with the Addition of Elemental Carbon. *Materials Letters*, **59**, 3795-3800. https://doi.org/10.1016/j.matlet.2005.06.032

[8] Selcuk, C. and Kennedy, A.R. (2006) Al-TiC Composite Made by the Addition of Master Alloys Pellets Synthesized from Reacted Elemental Powders. *Materials Letters*, **60**, 3364-3366. https://doi.org/10.1016/j.matlet.2006.03.021

[9] Birol, Y. (2008) Response to Thermal Exposure of the Mechanically Alloyed Al/C Powder Blends. *Journal of Alloys and Compounds*, **460**, L1-L5. https://doi.org/10.1016/j.jallcom.2007.05.071

[10] Karantzalis, A.E., Wyatt, S. and Kennedy, A.R. (1997) The Mechanical Properties of Al-TiC Metal Matrix Composites Fabricated by a Flux-Casting Technique. *Materials Science and Engineering*: *A*, **237**, 200-206. https://doi.org/10.1016/S0921-5093(97)00290-6

[11] Tong, X.C. and Fang, H.S. (1998) Al-TiC Composites *in Situ*-Processed by Ingot Metallurgy and Rapid Solidification Technology: Part II. Mechanical Behavior. *Metallurgical and Materials Transactions A*, **29**, 893-902. https://doi.org/10.1007/s11661-998-0279-7

[12] Nutt, S.R. and Carpenter, R.W. (1985) Non-Equilibrium Phase Distribution in an Al-SiC Composite. *Materials Science and Engineering*, **75**, 169-177. https://doi.org/10.1016/0025-5416(85)90187-9

[13] Banerji, A. and Reif, W. (1986) Development of Al-Ti-C Grain Refiners Containing TiC. *Metallurgical Transactions A*, **17**, 2127-2137. https://doi.org/10.1007/BF02645911

[14] Rapp, R.A. and Zheng, X. (1991) Thermodynamic Consideration of Grain Refinement of Aluminum Alloys by Titanium and Carbon. *Metallurgical and Materials Transactions A*, **22**, 3071-3075. https://doi.org/10.1007/BF02650269

[15] Yokokawa, H., Sakai, N., Kawada, T. and Dokiya, M. (1991) Chemical Potential Diagram of Al-Ti-C System: Al_4C_3 Formation on TiC Formed in Al-Ti Liquids Containing Carbon. *Metallurgical Transactions A*, **22**, 3075-3076. https://doi.org/10.1007/BF02650270

[16] Jarfors, A., Fredriksson, H. and Froyen, L. (1991) On the Thermodynamics and Kinetics of Carbides in the Aluminium-Rich Corner of the Al-Ti-C Phase Diagram. *Materials Science and Engineering*: *A*, **135**, 119-123. https://doi.org/10.1016/0921-5093(91)90547-Z

[17] Svendsen, L. and Jarfors, A. (1993) Al-Ti-C Phase Diagram. *Materials Science and Technology*, **9**, 948-957. https://doi.org/10.1179/mst.1993.9.11.948